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REDUCED TOXICITY, HIGH PERFORMANCE MONOPROPELLANT AT THE U.S. AIR FORCE RESEARCH LABORATORY

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ABSTRACT

Current programs are aiming to develop reduced toxicity monopropellant formulations to replace spacecraft hydrazine monopropellant. The Air Force Research Laboratory's (AFRL's) approach to replacing hydrazine is the synthesis and development of energetic compounds/formulations with substantially less vapor toxicity and superior performance (specific impulse and density). Characterization and testing of these high energy density materials is an essential part of the screening process for viable advanced propellants. Hazardous handling characteristics, undesirable physical properties or unacceptable sensitivity behaviors must also be identified and/or modified to further development by a potential user.

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1. INTRODUCTION

Due at least partially to the simplicity of system design, monopropellant system development has been an enduring subject of aerospace research and development. During the 1940s and 1950s efforts focused on evaluations of monopropellants such as hydrogen peroxide, propyl nitrate, ethylene oxide and hydrazine. The Jet Propulsion Laboratory (JPL) championed the use of hydrazine in Voyager in the 1970s and hydrazine eventually has become the monopropellant of choice for small engines of spacecraft in attitude, on-orbit maneuvering and gas generator applications.¹

The high vapor toxicity and large vapor pressure of hydrazine, coupled with the desire to both improve operational response and significantly increase

performance, present significant technical challenges to be overcome in producing next-generation monopropellants. The approach taken by the Air Force Research Laboratory is the use of energetic ionic compounds to replace hydrazine. In the past such efforts often attempted to produce low melting point salt mixtures containing toxic hydrazines and amines as melt point depressants.² JPL, the Naval Ordnance Testing Station, Naval Research Laboratory and other laboratories have examined such mixtures of salts and solvents since the 1950s. This work arose from efforts to find a hydrazine replacement with a significantly lower melting point. Typically, mixtures of hydrazine with its salts tended to be too detonable. Also, the British examined mixtures of ammonium nitrate, a fuel and water. Such compositions usually suffered from poor performance.²

Energetic ionic compounds can produce low melting point, liquid mixtures (especially in combination with a melting point depressant such as water) suitable as monopropellants. The coulombic attraction of these ions acts to tightly hold them in the liquid phase and consequently reduces the risk of toxicity posed by the vapor of the monopropellant. This mechanism of vapor pressure reduction has been generally recognized for salts such as ionic liquids.³

It is interesting to note two major efforts that have occurred over the last 15-years and have focused on advanced monopropellant compositions produced from energetic ionic compounds- one effort coming from Europe (specifically Sweden) and the other coming from the USA (specifically US Air Force). While the European monopropellant is based upon ammonium dinitramide, the AFRL effort expressly focuses on non-ADN propellant composition. While a significant amount of literature exists on the characteristics of the European monopropellant, essentially no literature is available regarding AF-M315E.⁴ This paper addresses the general safety and handling characteristics of AF-M315E which have led the US Air Force to conclude that this propellant combines both high performance with exceptionally low hazard potential.

Table 1. Desirable Monopropellant Small-Scale Safety Properties

Characteristic	Objective
Thermal stability	< 2% by wt. decomposition for 48 hrs at 75 °C
Unconfined ignition response	No explosive response
Impact sensitivity [Olin Mathiesen drop weight]	>20 kg-cm minimum
Friction sensitivity [Julius Peters sliding friction]	Insensitive at high load ($\geq 300N$)
Detonability [NOL card gap]	Class 1.3; (Zero-Card)
Adiabatic compression [U-Tube test]	Insensitive (Pressure ratio of 35)
Electrostatic discharge sensitivity	Insensitive to static spark discharge (1J)
Vapor toxicity	Low hazard (No SCBA requirement)

2. IHPRT SPACE PROPULSION DEVELOPMENT

The AFRL has pursued spacecraft chemical propulsion under aegis of the Integrated High Payoff Rocket Propulsion Technology (IHPRT) program since 1995. One goal set forth by the program is to develop a propulsion system with a 50% increase in density impulse over hydrazine employing a lower toxicity, advanced monopropellant. After assessment of several propellant approaches, one propellant was identified as most suitable for continued development in 2001- AF-M315E. This propellant incorporates energetic, low melting point salts (i.e., ionic liquids) as the principal components. Significant collaborative efforts from US Air Force, US Navy, Missile Defense Agency, Atlantic Research Corporation, General Dynamics, AMPAC, and GenCorp-Aerojet have characterized the formulation. The results of this effort have supported the selection of an AF-M315 propellant for ensuing IHPRT thruster demonstration efforts. The following sections outline the outstanding characteristics of AF-M315E.

3. MONOPROPELLANT SMALL-SCALE HAZARD PROPERTIES

There are a number of properties that are desirable for a monopropellant successor to hydrazine. Table 1 outlines eight objectives to be met for an acceptable monopropellant. These are safety and hazard properties that would determine suitability of a propellant to move forward to larger scale tests. Consequently, this listing should not be taken as comprehensive set of end-use criteria. Property requirements are mission dependent and can certainly be more exacting in nature. Other characteristics which are important to consider for propellant evaluation and use include vapor pressure, viscosity as a function of shear rate and temperature, surface tension, compatibility with hardware materials, propellant cost, ignitability, combustion temperature and combustion behavior over the applicable engine chamber pressure range. Table 1 represents AFRL's first level set of success criteria, and it should also be noted that many of these criteria and requirements are

derived directly from the US Department of Defense Ammunition and Explosives Hazard Classification Procedures TB 700-2.⁶

3.1 Thermal Stability

Determination of thermal stability was conducted via thermogravimetric analysis in which the weight loss of propellant sample (minus any inert volatiles) is monitored as a function of time. Average weight loss was determined to be 0.86 wt% over a period of 48 hours at 75°C, and negligible self-heating of the sample was observed. This finding demonstrated acceptable small-scale thermal stability in accordance with TB 700-2.⁶

3.2 Unconfined Ignition Response

The response of multiple unconfined quantities (154-g) of propellant placed into a wood-fueled fire was evaluated according to standard protocol TB 700-2. The propellant underwent a mild burning reaction with no explosive reaction. Consequently the propellant is judged to have a satisfactory hazard response in this small scale ignition response test.

3.3 Impact and Friction Sensitivity

Both impact and friction sensitivity tests were performed with the methodology specified in protocol TB 700-2. In regard to friction sensitivity the propellant is largely insensitive- showing negative reaction near the highest setting of the Julius Peters Testing equipment (i.e., negative response at 300N). The Olin-Mathieson impact sensitivity of the propellant was found to be 126 kg-cm (E_{50}) with a 3 kg drop weight. Thus, both friction and impact sensitivity properties of the propellant meet the objectives in Table 1 and pass the requirement set by TB 700-2.

3.4 Detonability and Critical Diameter

Resistance to detonation is a prime characteristic that a spacecraft propellant should possess. The Gap Test for detonability was conducted in accord with procedures outlined in Test Series 1 and 2 of TB 700-2. In this test the propellant is placed into a stainless steel cylinder and subjected to the full blast impact of a booster charge of Pentolite (or a composition of TNT and wax at 95/5 wt/wt, respectively). The condition of the test witness plate was examined to determine whether the propellant sustained a detonation through the length of the containment pipe. The results clearly showed the propellant was not susceptible to detonation in strong confinement at a diameter of 3.65-cm inner diameter. Thus, the critical diameter was certainly greater than the diameter of the containment cylinder, and larger scale testing would be pursued (see Section 5) to further gauge the critical diameter of AF-M315E.

3.5 Adiabatic Compression Sensitivity

An AFRL apparatus was used for assessment of the sensitivity of the propellant toward mechanical shocks (Adiabatic Compression Tester). The propellant sample is placed into a 316 stainless steel U-tube, and the sample is then exposed to an abrupt mechanical shock produced by the rapid introduction of nitrogen gas into the tube at a pressure between 3.45 and 20.7 MPa (500 to 3000 psi). A pressurization rate of 827 MPa/sec (120,000 psi/sec) was measured for the apparatus operated at 20.7 MPa driving pressure. The propellant was equilibrated to 25°C and the nitrogen pressure was 3.45 MPa for a driving pressure ratio of 35/1. A positive reaction of the propellant to adiabatic compression results in a deformed and possibly fragmented steel tube. Tests were performed and hydrazine was employed as a test control. The propellant displayed 9 consecutive negative reactions (no U-tube deformation) at a driving pressure ratio of 35/1 at 25°C. The loss of a tube end cap was noted in one test. The test on hydrazine also resulted in a negative response. Consequently, AF-M315E is found to be relatively insensitive to adiabatic compression under the applicable test conditions.

3.6 Electrostatic Discharge (ESD) Sensitivity

The response of AF-M315E to inadvertent ignition via a spark discharge was tested using an ESD test apparatus designed by the US Navy. Starting with sparks at energies of 0.25J, the propellant is exposed to increasingly greater spark energies to determine the threshold at which a burning or explosive response is observed. The propellant was found to be ESD insensitive, yielding 10 consecutive negative responses at spark energies of 1.0J.

In summary, AF-M315E possesses acceptable, in some cases outstanding, small-scale safety and hazards properties for thermal stability, unconfined burning response, impact and friction sensitivity, adiabatic compression sensitivity, ESD sensitivity and detonability. The important aspect of toxicology of AF-M315 is addressed in the following section.

4. TOXICOLOGICAL ASPECTS OF AF-M315E

Since the US Air Force desires the eventual approval of AF-M315E for missions that can require the close proximity of personnel, there are key questions regarding the relative toxicity of the propellant that must be answered. For this effort, initial toxicity testing of the propellant consisted of the following acute studies: Dermal Irritation, Dermal Sensitization, Oral Toxicity, and the Bacterial Reverse Mutation test. Results of these tests for AF-M315E were also compared with hydrazine. A study was also performed to investigate whether toxic compounds existed in the propellant's vapor phase at ambient and elevated temperature.

4.1 Acute Oral Toxicity

An acute toxicity study of AF-M315E administered by the oral (gavage) route to rats (Up/Down Design) was conducted. The purpose of the study was to assess the short-term toxicity of AF-M315E when administered by a single oral dose to rats. Based on the OECD 425 Acute Oral Toxicity Statistical Program with the default sigma of 0.5, the dose level increased or decreased (175 mg/kg, 550 mg/kg, and 1750 mg/kg) as each level was tested. The following variables and end points were evaluated in this study: clinical signs, body weights and gross necropsy. No significant gross internal findings were observed among lower dose animals at necropsy on final day (Day 14) of the test regime. The acute oral LD₅₀ of AF-M315E was estimated to be 550 mg/kg in the female rat with a 95% PL confidence interval of 385.3 to 1530. Since the oral LD₅₀ of hydrazine is 60 mg/kg in the rat, AF-M315E is an order of magnitude less toxic by the acute oral route.

4.2 Acute Skin Irritation

An acute skin irritation study of AF-M315E was pursued through administration by the dermal route to rabbits. The purpose of this study was to assess the irritant and/or corrosive effects of AF-M315E when administered by a single dermal dose to rabbits. Exposure to the propellant produced very slight erythema at 1/3 of the test sites by the 1-hour scoring interval. Complete resolution of the dermal irritation occurred by 72 hours post-dose. Under the conditions of the test, AF-M315E is considered to be a slight irritant to the skin of the rabbit based on the EPA-

FIFRA Dermal Irritation Descriptive Classification. The calculated Primary Irritation Index for the test article was 0.25. According to the European Economic Community (EEC) Dermal Evaluation Criteria, AF-M315E is considered to be a nonirritant for erythema and edema. In comparison, hydrazine is considered such a strong irritant that it is labeled as corrosive.

4.3 Dermal Sensitization

A sensitization study was undertaken for AF-M315E administered by the dermal route using the Modified Buehler Design. The dermal sensitization potential of AF-M315E was evaluated in Hartley-derived albino guinea pigs. An α -hexylcinnamaldehyde (HCA) positive control group consisting of 10 HCA test and 10 HCA control guinea pigs was included in this study to assure test fidelity. Based on the results of this study, AF-M315E is not considered to be a contact sensitizer in guinea pigs. Hydrazine by comparison is a strong allergen or potent sensitizing agent. The results of the HCA positive control study demonstrated that a valid test was performed for AF-M315E and indicated that the test design would detect potential contact sensitizers.

4.4 Ames Test for Mutagenicity

An Ames evaluation of AF-M315E for mutagenic activity was conducted using *Salmonella-Escherichia coli* microsome plate incorporation assay. Control values were consistent with historical values for this study. AF-M315E was judged to be mutagenic under some test conditions used in this study; therefore, the test substance was determined to be positive in the bacterial reverse mutation assay. It should be stated that it is exceedingly difficult to develop an advanced propellant that will not possess some mutagenic potential because of the chemical structures involved for energetic materials. Although AF-M315E is identified as mutagenic, it appears to be a weak mutagen compared to the potent mutagen, hydrazine. Advanced energetic propellant compositions that have been championed for environmentally enhanced or ‘green’ propulsion have included mutagens – examples include hydrazinium nitroformate (HNF) or ammonium dinitramide (ADN).⁷ AF-M315E is not an exception in this case.

The overall toxicological character of AF-M315E is such that the handling of the propellant does not require the self contained breathing apparatus (SCBA) that is required with handling of hydrazine. Instead, typical personal protective equipment (i.e., gloves, eye protection, and overalls/coats) is required. Consequently, the expenses of toxic release monitoring, equipment maintenance, and training for crews is eliminated.

5. LARGER SCALE HAZARD CHARACTERISTICS

The Air Force Research Laboratory (AFRL) has performed a variety of tasks to further characterize and assess the larger scale hazards of AF-M315E. The scope of the program encompassed evaluating the response of AF-M315E in a heavily confined vessel to thermal insult (e.g., slow cook-off) and obtaining a substance final hazard classification (FHC) for the packaged propellant.

An FHC is recognized to facilitate the transition of the propellant to the user community and address range safety requirements for potential flight demonstrations. For nearly a decade AFRL has been transporting AF-M315E under an interim hazard classification of 1.3C (shipped in 5 gallon composite pails) to support several DoD programs. As the propellant has advanced in development, it has become necessary to obtain an FHC for the mature propellant. In August of 2008, the US DDESB and the Joint Hazard Classifiers approved a substance FHC test plan consisting of an external fire test and stack tests on the packaged propellant consisting of a 5 gallon pail overpacked in a 20 gallon drum. However, additional tests such as the Super Large Scale Gap Test (SLSGT) and several single package tests were performed to supplement the data from the FHC testing.

5.1 Slow Cook-off

Initial confined slow cook-off tests began with a heavily instrumented 2.54-cm diameter sub-scale slow cook-off test developed jointly by AFRL and US Navy specifically to address the needs of this program. The full-scale tests included a 17.8-cm diameter vessel to help encompass a range of vessel sizes for spacecraft. The test sequence consisted of quickly heating the pipe at 10 C/min to 60°C for a three hour soak time at which point the temperature was ramped at 3°C/hour until thermal runaway and catastrophic failure occurred. The pipes contained 50 grams of propellant. All three ruptured in the center as evidenced by the fragments and high speed photography. The reaction violence was consistently mild with only a few large fragments thrown for each test. The onset of irreversible, exothermic reaction however, was observed at about 140°C in all of the tests.

The 17.8-cm diameter test used a 304 Stainless Steel pipe 16.5-cm in inner diameter with 0.64-cm thick walls. The center of the pipe was then machined to provide a theoretical burst pressure of 34.5 MPa (5000 psi). On each end a 15.2-cm long cylindrical piece of Teflon was used to insulate the heated section from the end caps. Next, a 61-cm long probe with 13

thermocouples spaced 5.1-cm apart was installed internally along the center axis from the bottom. The pipe was filled with slightly over 11.3-kg of propellant to allow for 25% ullage. A 68.9 MPa pressure transducer was then installed above the gas phase to attempt to determine the burst pressure. The entire article was then placed in a plywood box and heated in the same manner as the smaller 2.54-cm diameter tests with the exception of additional external thermocouples. The temperature controller thermocouple at failure was reading approximately 146°C, the same temperature as demonstrated in the 2.54-cm sub-scale tests. The test lasted nearly 37.9 hours before the pipe catastrophically failed towards the center of the pipe as desired. The reaction was violent, however the fragment evidence indicates this was not a detonation.

5.2 External Fire Test

As part of the FHC task, an external fire test was conducted in accordance with US MIL-STD-2105C in the configuration described in the substance FHC test plan submitted to the USAF Hazard Classifier. The unit configuration was comprised of six composite shipping pails (22.7-L, 5 gal, each) of AF-M315E totaling 163-kg (360-lb) of propellant. The pails were banded together on a metal grid with a fuel fire beneath to assess the type of reaction and reaction violence. The fuel fire burned for approximately 30 minutes, during which six distinct “pops” were audible starting at approximately 1 minute into the test. Each “pop” was associated with the lid of the steel overpack breaking away from the seal clamp around the top of the pails. Further into the test at approximately 5, 6, 7 and 8 minutes, propellant burns flared up within the fuel fire. The pre- and post-test images of the test article showed the configuration intact with very little damage to the outside packaging other than deformed lids. All of the propellant was consumed during the test along with the inner plastic propellant container. There were no obvious fragments thrown or any evidence of blast overpressure (Bikini gauges were unchanged). Overall, the propellant exhibited a mild burning (i.e., Type V) reaction, which was an excellent response in large fire situations.

5.3 Single Package Tests

A series of single package tests were performed to identify useful packaging materials and configurations. Two configurations (Configurations A and B) were explored and tested in triplicate. All tests were conducted with the plastic explosive (C4) donor placed in the center of the outer container lid. The first packaging configuration, Packing Configuration A, consists of a 22.7-L, 5 gal, composite pail (DOT/UN 1A2/X50/S), overpacked in a 90.9-L ,20 gal, thin-wall steel drum (UN 1A2/X220/S, UN 1A2/Y1.5/150

Removable Head Steel Drum) with the space between the inner pail and the drum filled with a combination of sand and polyurethane foam packing material.

The second packaging configuration, Packaging Configuration B, consists of a 4.5-L F-Style plastic jug overpacked in a 22.7-L plastic pail (1H2/Y25/S/09). For transportation and storage purposes this plastic jug is wrapped in a polyethylene plastic bag. The space between the jug and the pail is filled with a combination of sand and an appropriate compatible polymeric packing material to brace the inner container.

The two assembled configurations identified above were subjected to a series of single package detonability tests. Each article, including the 90.0-L drum tests, was placed on a 0.32-cm thick 61x61-cm witness plate. Each test was initiated by a 0.11-kg (1/4 lb) C4 donor charge placed in the center of the lid of the container or drum and initiated by a #8 strength blasting cap. No fragment collection was performed as a requirement of the test, but observations were made as to the fragment size and disbursement from each test. Three tests were conducted on Packaging Configuration A. Initiation was again with the 0.11-kg C4 donor placed on the center of the lid, directly over the inner poly container with the liquid propellant. All three 90.9-L package tests resulted in almost identical, non-detonating reactions. Typical of the reactions was a hole punched through the drum lid by the donor explosive resulting in a plume of package material and liquid propellant being expelled upward from the drum. Inspection of the damage through the hole revealed that most of the sand was still over the inner pail. None of the inner plastic containers were broken or damaged to the point that they leaked any liquid propellant into the outer drum.

Three tests were also conducted on Packaging Configuration B. All three tests resulted in near identical non-detonating reactions. Typically, the outer pail is split in several places and the inner sample jug is peeled or folded open. The sand and packaging materials are strewn about the test area and most of the liquid propellant is splattered over a 9.1-m (30 foot) diameter area. No damage or bending of the witness plate occurred in any of the three tests in this configuration. The blast overpressure measured in all six tests was no greater than the overpressure caused by the C-4 donor further confirming no propellant contribution.

The excellent response of both package configurations to explosive shock has thus been demonstrated. Consequently, both types of packaging will be employed for AF-M315E.

5.4 Larger Scale Gap Tests

A 7 GPa (70 kbar) SLSGT with heavy confinement (1.27-cm steel walls) was conducted in-house concurrently with this program to better characterize the shock sensitivity of AF-M315E, and because this standardized test (TB 700-2, Section 5-8, Option 3) is increasingly being used to help differentiate between Class 1.1 and 1.3 propellants. The SLSGT test employs a 17.8-cm (7-in) inner diameter (20.3-cm (8-in) outer diameter), steel cylinder as the propellant containment, and its length is 81.3-cm (32-in). The testing was conducted using TB 700-2 as guidance in relation to test and sample set-up, booster material, booster/attenuator calibration, raw pin data, reaction velocity vs. pin distance plots, witness plate and recovered case projection photographs with video/film records, and blast gauge data provided. It should be noted that, in the case of rocket motors, to be classified Hazard Division/Class 1.3, the TB 700-2 requires the propellant to exhibit a decaying reaction approaching the velocity of sound in the propellant and meet the requirements of the External Fire test.

The outcome of the SLSGT test clearly demonstrated that AF-M315E passed the 7 GPa super large scale gap test as described by TB 700-2. The test results showed the propellant did not detonate as evidenced by a deformed witness plate and a measured shock speed that decayed to sonic velocity. Although this is a standardized test typically used to assess the shock sensitivity of large solid rocket motor propellants, it shows utility for liquid propellants as well and provides good supporting data for AF-M315E to be considered for a Hazard Division/Class 1.3 final hazard classification.

Gap tests were also conducted under strong confinement conditions and without booster blast attenuation at both 10.2-cm (4-in) and 17.8-cm (7-in) inner diameters. Under approximately 28 GPa blast pressure, the propellant failed to detonate at 10.2-cm, and a detonation was observed at 17.8-cm. Consequently, the critical diameter of AF-M315E can be stated to fall between 10.2-cm and 17.8-cm.

6. SUMMARY AND CONCLUSIONS

The very broad range of characterization of AF-M315E was aimed at answering key questions about the propellant's safety and hazard behavior and providing knowledge points to facilitate the transition of the propellant into fielded applications.

Overall, the results of the above efforts demonstrate that AF-M315E has attractive safety properties with verified reduced toxicity compared to the standard spacecraft

monopropellant, hydrazine. With respect to toxicity, it still appears that, with the exception of ingestion, the propellant poses little hazard and would require the minimum of PPE for handling. The formulation's vapor pressure is indeed negligible at room temperature with the exception of water.

The propellant behavior in small-scale cook-off scenarios (slow, fast, confined, and unconfined) was relatively mild. The 7" diameter full-scale confined slow cook-off test showed more reaction violence (not a detonation), but still had a good, high reaction temperature that was comparable to the sub-scale tests.

In hazard classification testing the propellant exhibited very benign reactions. Two package configurations were identified which showed no reaction to a C-4 donor shock stimulus. Consequently the US Department of Defense Joint Hazard Classifiers (JHC) and DDESB have verbally agreed to a HD Class 1.3C determination on the packaged substance and are processing the package for formal concurrence and signature.

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